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METHOD AND DEVICE FOR REMOVING MATERIAL FROM GAS

METHOD AND DEVICE FOR REMOVING MATERIAL FROM GAS

Patent Number: JP2001062244
Publication date: 2001-03-13
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Applicant(s): ASM MICROCHEMISTRY OY
Requested Patent: ☐ JP2001062244
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Priority Number(s):
IPC Classification: B01D53/34; B01D53/81; B01D53/68; B01J19/08
EC Classification:
EC Classification:
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Abstract

PROBLEM TO BE SOLVED: To provide a method and device for removing material from the gas discharged from a gas phase reactor.

SOLUTION: In the method for removing the material contained in the gas discharged from a ALCVD reaction process, the gas is brought into contact with the material having large surface area and kept in the substantially same condition with usual case in a gas phase reaction process, and the material having large surface area is exposed to surface reaction with the material contained in the gas to form solid reaction product on the surface of the material having large surface area and to remove the material from the gas. The amount of the waste generated at the gas phase process is reduced and the wear of equipment is reduced.

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Bibliography

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 (32) [Priority day] July 20, Heisei 11 (1999. 7.20)
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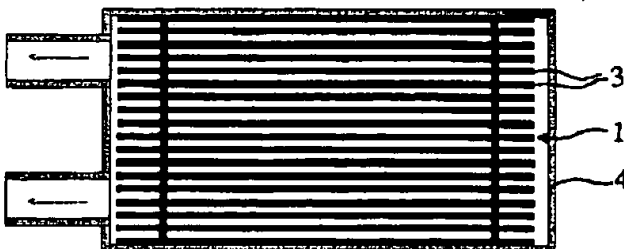
Summary

(57)

[Summary]

[Technical problem] Offer the technique and equipment for removing the matter
from the gas discharged from gaseous-phase-reaction equipment.
[Resolution means] Especially this invention is the technique for removing the
matter contained in the gas discharged from ALCVD reaction process. The
material which has the big surface area which usually comes out of a gas into a
gaseous-phase-reaction process, and was kept essential to the same conditions
as a certain thing is made to contact; And expose the material which has a big
surface area to the surface reaction with the matter contained in a gas, a solid-
state resultant is made to form on the front face of the material which has a big
surface area, and it is related with the technique containing removing the matter
from a gas. this invention decreases the amount of the waste produced in a
gaseous-phase process, and reduces consumption of a facility device.

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CLAIMS

[Claim]

[Claim 1] It is the technique for removing the matter contained in the gas discharged from a gaseous-phase-reaction process. The foam which has the big surface area which usually comes out of the overresponse object gaseous-phase pulse of ALD process into a gaseous-phase-reaction process, and was kept essential to some conditions and the same conditions is made to contact, And technique containing exposing the material which has a big surface area to the surface reaction with the matter contained in a gas, making a solid-state resultant form on the front face of the material which has a big surface area, and removing the matter from a gas.

[Claim 2] Technique given in the claim 1 which includes contacting the overresponse object gaseous-phase pulse of ALD process to a porous substrate in order to form a resultant on the surface of a material.

[Claim 3] Technique given in the claims 1 or 2 of which a foam consists including a porous graphite material, a porous ceramic, an alumina, a silica, and the material chosen from the group of glass wool.

[Claim 4] Technique given in any 1 term of the aforementioned claim which is the gas in which gaseous [a part of] contains a chloride at least.

[Claim 5] Technique given in any 1 term of the aforementioned claim to which the reaction with the material which has a gaseous-phase-reaction process and a big surface area is performed in the same reaction space.

[Claim 6] Technique given in any 1 term of the claims 1-4 by which the material which has a big surface area is placed into the individual reaction space connected with the reaction space of gaseous phase reaction.

[Claim 7] It is the equipment for removing the matter contained in the gas discharged from a gaseous-phase-reaction process. Consist of a reaction process including the reaction zone arranged down-stream, and have a big surface area, and essentially, in a gaseous-phase-reaction process, usually come out and it changes including a foam maintainable on some conditions and the same conditions. The aforementioned reaction zone is the equipment containing the gas passage for sending further the gas discharged from a gaseous-phase-reaction process into the material which has a big surface area, and the issue gas passage for discharging the material which has a big surface

area to a gas.
[Claim 8] Equipment given in the claim 7 to which a reaction zone is arranged inside the reaction shell of a reactor, and gaseous phase reaction is performed in reaction shell.
[Claim 9] Equipment given in the claim 7 arranged inside the individual reaction container with which the reaction zone was connected with gaseous-phase-reaction equipment, and was maintained by the same conditions.
[Claim 10] Equipment given in any 1 term of the claims 7-9 as which the material which has a big surface area is chosen from the group of a porous graphite material, a porous ceramic, an alumina, a silica, and glass wool.

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DETAILED DESCRIPTION

[Detailed description]
[0001]

[The technical field to which invention belongs] this invention relates to elimination of the matter contained in gases, such as a gas which flows by low voltage. Especially this invention relates to the technique and equipment for removing the unreacted reactant and steamy precursor which exist in the gas removed from gaseous-phase-reaction equipment.

[0002]

[Prior art] In an atomic-layer vacuum deposition (ALD), generally the substrate is located in reaction space and a substrate receives the surface reaction repeated by turns [of at least two kinds of different reactants]. ASM which has commercial technique in Espoo of Finland with a trademark called ALCVDTM Microchemistry It is supplied from Oy. According to this technique, a reactant is repeated and each reactant is put in into reaction space in the type of a gaseous-phase pulse from the source of itself by turns. A gaseous-phase-reaction object can react with a substrate front face here for the purpose of forming a solid-state thin film on a substrate.

[0003] Although this technique is the most suitable in order to make the so-called compound thin film as a reactant using such a start material containing the component of the compound thin film of a wish, it is also applicable to a growth element thin film (growing elemental thin films). In the compound layer currently

generally used by this technical field, ZnS layer adopted as electroluminescence display can be mentioned, zinc sulfide and a hydrogen sulfide are used as a reactant in a growth process in this case, and such a layer is grown up on a glass substrate. A silicon thin film can be mentioned in an element thin film.

[0004] ALD equipment contains the reaction space which can put a substrate on inside, and at least two sources of a reactant which can supply the reactant used in a thin film growth process in the type of a gaseous-phase pulse into reaction space. The source of a reactant is connected with reaction space through the reactant inflow way, and the outflow way (pump piping) is connected with the reaction space for being attached in a pump and removing the gas resultant of a thin film growth process, and the overresponse object of a gaseous phase.

[0005] Waste, i.e., the unreacted reactant which is removed from reaction space and discharged, is a serious problem for ALD treatment process. If this unreacted object enters into pump piping or a pump, monotonous and tedious depuration is needed, and in being the worst, it will wear out a pump quickly.

[0006] Although it becomes some support, law is both understood that it is inadequate for a long run to contact the thing for which a gas is filtered, and/or a gas to an absorbent. Since the waste which poses a problem does not contain easily the separation precursor of excessive amounts, such as water in which pump delivery is possible, a titanium chloride, or an aluminum chloride, as separation matter, it is not helpful to build heating formula pump piping expensive in order to remove waste through a pump. When the matter has reacted, the problem form in the interior of pump piping the by product which has low vapor pressure occurs. This problem is related, when a reactant reacts mutually at temperature lower than process temperature and an unsuitable reaction is triggered especially. At this temperature, an acid chloride may be formed as a by product. These by products form a lot of fine particles. Generally, this kind of reaction is generated between a reaction zone and the fraction with the low temperature of pump piping inside pump piping. Then, if the precursor which has high vapor pressure at a room temperature reaches a pump at the temperature suitable for thin film growth, another problem will occur. This may bring the result called laminating of the layer matter in the front face of a pump. It is very tended to wear the laminating of the formed matter out. This is a specific problem accompanied by heating formula pump piping and an elevated-temperature dry-type pump. narrow by this -- it will allow and ** will be closed, by this, parts will contact mutually and a pump will break The 3rd problem is a reaction between the steams of the pulse which follows the degree in the fraction and pump piping by which the preceded reaction component was condensed. This makes matter growth of CVD format, and a lot of fine-particles transmission cause.

[0007] As mentioned above, various solutions based on filtration or/of reaction waste, and a chemical preparation were tried in piping before a process for some dozens of years, and the result was a little bad. For the by product and fine particles which were formed tending to blockade a VCF, and maintaining at the status that a low process pressure is the cause and, as for the gas style, opened the mesh of a VCF, it is too weak. The blockaded VCF causes a failure of pressure further, therefore changes the matter style from a source. A process

pressure and a gaseous speed also change. In order to remove a by product from a mesh, use of a cyclone and a rotating type sublation machine was tried. According to these means, although a part of solid-state waste is removable, in addition, the precursor which has high vapor pressure will reach a pump, and will generate a by product there.

[0008] The Finland patent of No. 84980 (Planar International Oy) is indicating the system by which gaseous flowing consists of a condensation room which slows down a speed and the great portion of waste condenses. Since water excessive before going into a VCF unit is injected in VCF housing and the size of by-product grain is increased in order to prevent lock out of a VCF mesh, a rotating type sublation machine system removes waste. Although this equipment improves present condition technique clearly, it cannot yet be satisfied completely.

[0009]

[Object of the Invention] The purpose of this invention is offering the technical solution which it is [for eliminating the trouble of a Prior art and removing waste from the reaction zone of ALD reactor] easy a solution, and can be trusted.

[0010]

[The means for solving a technical problem] Before discharging a precursor from a reactor or a reaction zone, this invention processes all of the excessive precursors of the pulse dose, and is based on the idea of forming an end product. By this, the capacity of waste can be decreased sharply. After treatment for an overprecursor produced from ALD process is performed by arranging the material with a big surface area (generally porosity) in a reaction zone, and this material is paid by them while a precursor moves to the outlet of a reaction chamber. Or although it is the outside of a reaction zone, the material with a big surface area can be placed into the heated container with which it became independent before the drainage pump. However, in both the enforcement gestalt, it is kept essential on growth conditions (for example, the same pressure and temperature) as a reaction zone where, as for the matter with a big surface area, the material with a big surface area ensures growth of the resultant in a front face. Consequently, the material with a big surface area catches the remaining end product on the front face, and only a gas product is made to go into a pump.

[0011] The technique for removing the matter contained in the gas discharged from the gaseous-phase-reaction process by this invention, if furthermore specified The foam which has the big surface area which usually comes out of the overresponse object gaseous-phase pulse of ALD process into a gaseous-phase-reaction process mainly, and was kept essential to some conditions and the same conditions is made to contact, And expose the material which has a big surface area to the surface reaction with the matter contained in a gas, a solid-state resultant is made to form on the front face of the material which has a big surface area, and it is characterized by removing the matter from a gas.

[0012] Including the reaction zone arranged down-stream (namely, after) from the reaction process, a reaction zone has a big surface area, and this equipment essentially contains a maintainable material in effective conditions and the same

conditions among a gaseous-phase-reaction process. A reaction zone contains the gas passage for sending into the material with a big surface area further the gas discharged from the gaseous-phase-reaction process, and the exhaust passage for discharging a gas from the matter with a big surface area. [0013] The equipment for removing the matter contained in the gas discharged from the gaseous-phase-reaction process by this invention Consist of a reaction process including the reaction zone arranged down-stream, and have a big surface area, and essentially, in a gaseous-phase-reaction process, usually come out and it changes including a foam maintainable on some conditions and the same conditions. The aforementioned reaction zone is characterized by including the gas passage for sending further the gas discharged from a gaseous-phase-reaction process into the material which has a big surface area, and the issue gas passage for discharging the material which has a big surface area to a gas.

[0014] A remarkable advantage is acquired by this invention. For example, the material with a big surface area catches the end product of the reaction of a superfluous gas reactant on the front face. Generally the surface area of this trap is large, on the average, is about 10-1000m²/g, for example, can have the area of a football stadium, and the surface area of the same grade. A trap can be used several times, before washing or replacing with a new thing. It is only the gas-like matter which the pump connected with reaction space should cope with. It is because only the agenesis gas by product from a process reaches a pump. A solid-state thin film product is completely caught in a reactant trap, and this decreases consumption of a facility device remarkably.

[0015] Generally this invention is applicable to any gas reactants. This is advantageous to especially the reaction that forms the by product with detrimental others into the reaction of a gas reactant or it is corrosive. For example, the desirable 1 enforcement gestalt is for processing the waste which uses a chloride inclusion reactant like the aluminum chloride which reacts with water and generates a metallic oxide, and is generated by gaseous phase reaction. Although being used for ALD is desirable as for this invention, CVD process or conventional electron beam sputtering, and the discharged conventional gas reactant can also use it for processing the offgas from all the gaseous-phase processes of the others which act mutually behind a real reaction zone. However, this invention is specified as the ALD method and the following explanation indicates it. Next, this invention is considered still in detail with reference to the drawing of appending illustrating some alternative implementation gestalt.

[0016]

[Gestalt of implementation of invention] Generally this invention is based on the idea of placing the material which has the big surface area which forms the post-reaction substrate of the discharged excessive gaseous-phase-reaction object which leaves a real reaction zone immediately after the substrate of ALD reactor. An important thing is that the front face of the porous matter is so large that it is convertible for the last compound which corresponds according to the principle of ALD (atomic-layer vacuum evaporatio~~no~~) if all of excessive matter can stick to

the front face of a reactant trap and the following reactant pulse enters.
[0017] In a pyrogenetic-reaction zone, the reactant trap of a post-reaction can be placed into a vacuum housing, and even the space of a sink box can use it as an electrode holder for a trap receptacle.

[0018] An example about growth of the oxidation ***** layer by ALD technique is given. In aluminum₂O₃ process of 3000 cycles, H₂O of 100g of AlCl₃ 3 and 100g is consumed. 60g of 1/3 becomes aluminum 30g and 30g of oxygen about at aluminum₂O₃, and HCl of the amount whose 2/3 is 140g is formed. 1/3 with equal 20g of a precursor must be used for growth of the thin film product on a substrate, and 40g aluminum₂O₃ which is the 2/3 remaining must be caught by the trap. This means that about 40g [per run] solid-state is caught. aluminum₂O₃ which grew in each run has the thickness of 150nm, and this means that a 15-micrometer-thin film grows on a trap front face after 100 runs. The thin film which grew in the trap does not suppress a gas style by choosing the dimension and path length of pore so that all resultants can be swept away, before the following pulse goes into a trap so that there may essentially be no failure of pressure through a trap.

[0019] It is important that nothing is made to be about blockading the passage of the material which the acid chloride of a lot of big molecules arises, and has a big surface area absolutely.

[0020] According to this invention, if one or more trap blocks or two or more trap plates are materials large a surface area and suitable (for example, porosity), they can be made by every material (for example, graphite, an alumina (aluminum₂O₃), or silicas, such as a porous graphite foil). Mineral matter, such as various ceramic materials, for example, a honeycomb ceramic, and glass wool, can be used. Reticulated glassy carbon (Reticulated Vitreous Carbon) is other examples of a suitable material. The material needs physical and to be able to bear chemical conditions (reaction temperature and **) of a reaction zone (a material must be inactive at a reactant). Furthermore, in order to form resultants (aluminum oxide etc.), a material must have a big surface area so that the reaction of a gas reactant may be expected on the front face. Generally, the surface area of a trap material is 10 or 2000m²/g, and is about 100 or 1500m²/g especially. As one alternative, it permits that a gas reactant permeates into a material, and by products, such as a hydrochloric acid, are having the porous ceramic material which remains in a front face so that it can sweep away and which made the front face coarse. it is so narrow that the pore of a foam cannot sweep away the residue (unreacted) of a previous pulse before introducing the following pulse -- it must not elapse and be too deep averaging -- an about about 10-100-micrometer hole -- especially the pore that has a size is promising

[0021] According to this invention, it is still important that the front face of a reactant trap is so large that the same trap material can be used to growth of the number batch of two or more thin film elements. As inquired upwards, generally the amount of [of a reactant] luxus is 4 to 5 times the complement, in order to cover the front face of a substrate with the thin film of desired thickness. Therefore, the front face of a material must be at least 4 to 5 times as large as all the front faces of a substrate, and it is bigger and if it is ***** , there is nothing, so

that a front face may make continuous action possible all day long especially according to the production capacity of a reactor.

[0022] The further serious element of the material which has a surface area with a big reactant trap is that there must not be no essential pressure differential over the whole material. The passage which the material which has a big surface area by this ground makes free gaseous flowing possible, and offers sufficient gaseous-phase component for a front face for surface reaction must be given. The failure of pressure to generate shows various methods of attaining the free passage which becomes the minimum to the enforcement gestalt of a drawing.

[0023] If their eyes are turned to an attached drawing here, in drawing 1 a and drawing 1 b, it can regard that the reactant trap 1 (it can also be called an "afterburner" below) is put on the bottom of the real reaction space 2 of ADL reactor. A reactant trap grows into the interior of the sink box 4 of a reactor including two or more trap plates 3 each other placed in parallel. The passage formed so that a gas might flow on a pump further is between the trap plates 3. When the trap plate is made from the suitable material which has a big surface area, a reactant is made to deposit for the surface reaction similar to the reaction which diffuses a reactant gas inside a plate, for example, happens in the reaction space between a glass substrate and a reactant steam.

[0024] By arranging a reactant trap directly behind a reaction zone or to the bottom, the free flowing path or free passage for an overresponse object can be prepared easily. Similarly, since the lowered pressure which this gas produces by the same drainage pump as the remainder pressure of a reactor is received, it is easy to discharge the gas from a reactant trap.

[0025] After sending [be / under / of reaction space / continuation / it] each reactant pulse into the reactant trap 1, generally reaction space is swept by inactive or inactive gas, such as nitrogen. And the following gaseous-phase pulse is sent in into reaction space (and reaction trap). ** -- like, after an aluminum chloride pulse, a steam pulse is usually sent in into reaction space, and an aluminum chloride is changed into an aluminum oxide. The same reaction arises in a reactant trap the front-face top of the substrate placed into ALD reactor. By putting a reactant trap on the interior of the same reaction space as a substrate, or a reaction box, temperature level required in order to attain ALD (atomic-layer vacuum evaporation) reaction on the front face of a trap material, and pressure level are obtained automatically. A reactant forms the same end product, for example, ATO, or aluminum₂O₃ also on a substrate on the front face of a trap.

[0026] Except for the enforcement gestalt of drawing 2 a and drawing 2 b being placed into the different container 13 with which the reactant trap 11 was maintained at the same reaction condition as a reactor, it is similar with the above-mentioned enforcement gestalt. Although the trap plate 12 is arranged in the format similar to the plate in drawing 1 a and drawing 1 b, passage is arranged so that the path which wound may be offered. By this technique, sufficient contact time with a trap plate can be offered. The reactant trap container is attached in the sink box of ALD reactor by the closed conduit (conduct).

[0027] The plate is being fixed to the passage and the parallel relation between

them although it corresponds to what the enforcement gestalt of drawing 2 and drawing 3 combined in the meaning of being placed into the container 23 with the trap plate 22 separate [the enforcement gestalt of drawing 3 a and drawing 3 b]. The plate is made from glass wool.

[0028] Drawing 4 a and drawing 4 b show the exchange formula cartridge 32 which has the passage 33 which was made from the material which has big surface areas, such as glass wool (drawing 4 a), and was formed into this material. The same passage 35 is arranged between the adjacent layers of the graphite foil 34 wound around the whorl format in drawing 4 b. A mutual spacing is preferably arranged by about 0.5-5mm about 0.1-10mm, and these layers have desirable things.

[0029] The trap of drawing 4 a and drawing 4 b is made from such a cheap material after the effective duration of service that it can discard.

[0030] With all the enforcement gestalt from drawing 2 to drawing 4, the operation of a precursor trap resembles very much what was explained about the enforcement gestalt of drawing 1 a and drawing 1 b. The material which has a big surface area is maintained by the same temperature (namely, responding to a precursor and a substrate about 50 or 600 degrees C, generally about 200 or 500 degrees C) as the thing of a real reaction zone. Although a pressure can be made into atmospheric pressure, it is desirable to operate by the pressure (namely, "low voltage") to which about one to 100 hPa (mbar) generally decreased. The inactive gas used for eradication elimination changes including rare gas, such as nitrogen or an argon.

[0031] Please regard that it is applicable to all the chemistry gas vacuum evaporatio used, for example for manufacture of the catalyst in a gaseous phase although the above-mentioned enforcement gestalt is divided and is related for manufacture of the diaphragm structure on all kinds of front face in a semiconductor device and flat-surface panel equipment.

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DESCRIPTION OF DRAWINGS

[An easy explanation of a drawing]
 [Drawing 1 a] In order to form a reactant trap into the sink box of ALD reactor, it is the schematic diagram seen from the upper part which shows how to absorb a

porous plate and put on the interior of a box.
[Drawing 1 b] In order to form a reactant trap into the sink box of ALD reactor, it is the side elevation showing how to absorb a porous plate and put on the interior of a box.

[Drawing 2 a] It is drawing showing arrangement of the plate in the interior of the separate back reactor connected with the sink box of ALD reactor.

[Drawing 2 b] It is the same drawing as drawing 2 a which shows arrangement of the plate in the interior of the separate back reactor connected with the sink box of ALD reactor.

[Drawing 3 a] It is drawing different in that the plate was transposed to the cartridge of glass wool corresponding to drawing 2 a.

[Drawing 3 b] It is drawing different in that the plate was transposed to the cartridge of glass wool corresponding to drawing 2 b.

[Drawing 4 a] It is the cross section of a cartridge filled with glass wool.

[Drawing 4 b] It is the cross section of a cartridge filled with the graphite foil.

[Translation done.]

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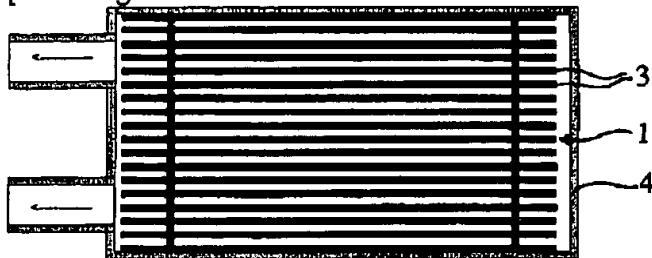
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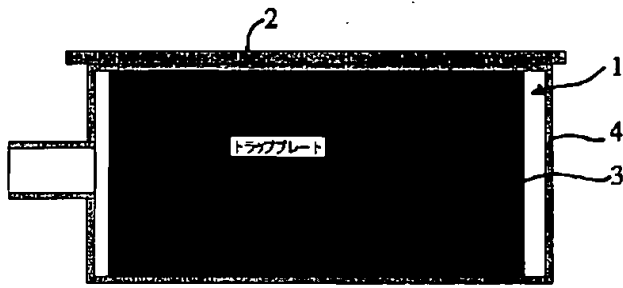
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DRAWINGS

[Drawing 1 a]



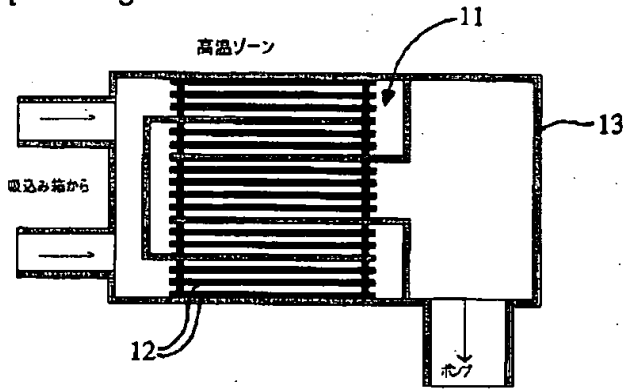
[Drawing 1 b]



[Drawing

2

a]



[Drawing

2

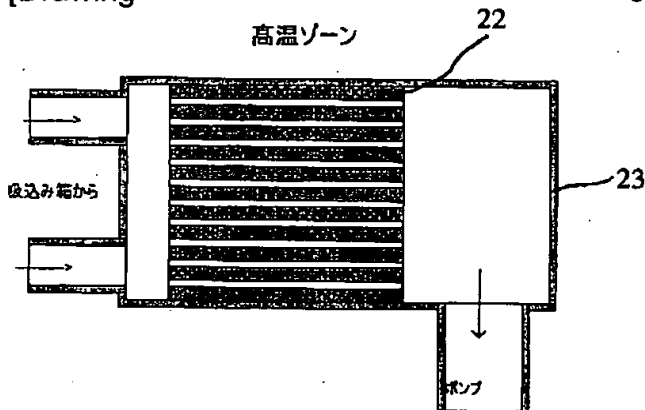
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[Drawing

3

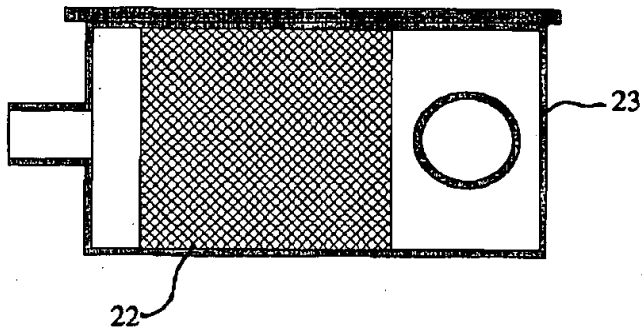
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[Drawing

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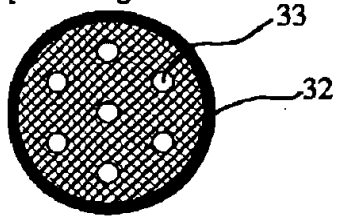
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[Drawing

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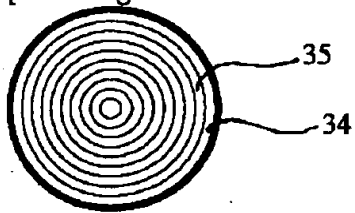
a]



[Drawing

4

b]



[Translation done.]

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